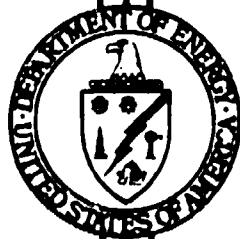


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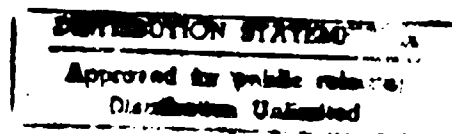
## LOW TEMPERATURE DEGRADATION OF Y-TZP MATERIALS

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## ABSTRACT

Seven yttria-tetragonal zirconia polycrystal materials were examined to determine if they were susceptible to low temperature degradation. Flexure specimens were exposed to -800 Pa of water vapor pressure for 50 hours between 200°C and 400°C. Only one of the TZPs was unaffected by these low temperature treatments; three underwent catastrophic degradation after all the low temperature treatments, while the final three had property losses to various degrees depending on the treatment temperature.

Keywords: High Strength, Toughness, Ceramics,  
microstructure, Transformation (15H)

## FOREWORD

The work described herein is part of the Characterization of Transformation Toughened Ceramics Program which is a subtask of the Department of Energy (DOE) sponsored, Oak Ridge National Laboratory (ORNL) monitored, Ceramic Technology for Advanced Heat Engines Project, (Interagency Agreement No. DE-AI05-84OR21411). The purpose of this subtask is to examine commercial and experimental transformation-toughened ceramics for possible application in advance heat engines.

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## CONTENTS

	Page
FOREWORD . . . . .	iii
INTRODUCTION . . . . .	1
EXPERIMENTAL PROCEDURE . . . . .	2
RESULTS	
Observations . . . . .	3
Density and Modulus of Elasticity . . . . .	6
Phase Stability . . . . .	7
Strength . . . . .	7
Fractography . . . . .	8
DISCUSSION . . . . .	15
CONCLUSIONS . . . . .	16
ACKNOWLEDGEMENT . . . . .	17
REFERENCES . . . . .	17
APPENDIX: ZIRCONIA DATA AFTER LOW TEMPERATURE EXPOSURE . . . . .	18

## INTRODUCTION

Yttria-tetragonal zirconia polycrystal (Y-TZP) materials are candidates for structural applications because of an unusual combination of high strength and toughness. These excellent properties stem from a stress-assisted "martensitic" transformation of a metastable tetragonal (t) phase to the stable monoclinic (m) phase. It is believed that absorption of the crack tip energy by the t-grains in the vicinity of the crack tip causes the transformation resulting in enhanced properties. Other mechanisms, such as crack tip deflection and microcracking, may also contribute to this enhancement.

Since Kobayashi et al.<sup>1</sup> first reported the degradation of  $\text{ZrO}_2\text{-Y}_2\text{O}_3$  polycrystalline materials at low temperatures (150°C to 400°C), many additional studies<sup>2-17</sup> have been initiated to further examine this phenomenon. Their general observations can be summarized as follows:

- The degradation is most pronounced between 200°C and 300°C.
- The degradation results in large decreases in strength, toughness, and density, and a significant increase in the m- $\text{ZrO}_2$  content.
- The degradation is due to the  $t \rightarrow m$  transformation which is accompanied by micro- and macrocracking.
- The  $t \rightarrow m$  transformation initiates on the surface and proceeds into the bulk.
- A decrease in grain size and/or an increase in the stabilizer content slows the transformation.
- The presence of water or water vapor greatly enhances the transformation.

Theories based on the interaction between the  $\text{ZrO}_2\text{-Y}_2\text{O}_3$  and  $\text{H}_2\text{O}$  have been put forth to explain the rapid, low-temperature degradation of Y-TZP materials. Sato and Shimada<sup>5</sup> have proposed a theory where Zr-OH bonds are formed when water reacts with Zr-O-Zr bonds at the crack tip. They have shown that in addition to aqueous solutions, certain nonaqueous solutions with a lone-pair electron orbital opposite a proton donor site enhance the rate of  $t \rightarrow m$  transformation at low temperatures. It was concluded that under these conditions the strain which acts to stabilize the tetragonal phase may be released and this, coupled with the growth of preexisting flaws, accelerates the transformation.

The hypotheses of Yoshimura et al.<sup>10,13</sup> is similar in that it is also based on the formation of Zr-OH bonds, but they suggest a four-step degradation process. The  $\text{H}_2\text{O}$  is absorbed onto the Y-TZP surface and dissociates forming Zr-OH bonds which creates stress sites. The stresses build as the  $\text{OH}^-$  ions diffuse through the surface and the lattice resulting in "nucleation" sites for the phase transformation. When the stress buildup reaches a critical level, the  $t \rightarrow m$  transformation will occur with the associated micro- and macrocracking enabling the process to continue into the bulk. Their assumptions are based on findings which show that the transformed m- $\text{ZrO}_2$  has an  $\text{OH}^-$  group which is not found in the untransformed t- $\text{ZrO}_2$ , the addition of the  $\text{OH}^-$  group to the monoclinic phase increases the lattice parameter, and the lattice parameter can be returned to its original value if the specimen is dried.

Lange et al.<sup>7</sup> have seen the formation of 20 nm to 50 nm crystallites of  $\alpha\text{-Y}(\text{OH})_3$  and proposed that the formation of this hydroxide draws yttria from the tetragonal grains on the surface creating monoclinic nuclei. Growth of the nuclei continues with further yttria depletion until it reaches a critical size where it can spontaneously grow, transforming the tetragonal grains into

monoclinic. If the transformed grains are large enough, microcracking will occur. The microcracking provides an avenue for the water to penetrate to subsurface grains allowing the process to repeat. If the transformed grains are smaller than the critical size required for microcracking, then the transformation and subsequent degradation will be limited by the long-range diffusion of yttria to the surface. Work by Winnubst and Burggraaf,<sup>8</sup> using Auger electron spectroscopy, has shown a homogeneous yttrium-rich surface layer on specimens after only five hours of exposure to 177°C in a nitrogen environment. This clearly supports the theory of Lange and his co-workers.

The theories reviewed here are based on observations and, at this time, are still speculative. Other potential theories should not be ruled out, and further work is still needed to detail the mechanisms of this low-temperature degradation.

The purpose of this study was to determine whether several commercial and experimental Y-TZP materials are affected by low-temperature annealing in the presence of water vapor.

### EXPERIMENTAL PROCEDURE

Y-TZP materials were obtained from a variety of manufacturers which are listed in Table 1. Type "B" bars (3 mm x 4 mm x 50 mm) were machined from billets of these materials according to MIL-STD-1942 (MR). Due to material limitations, the AC Sparkplug TZP-110 was machined into "A" (1.5 mm x 2 mm x 30 mm), as well as type "B" bars, according to the same standard. The bars were then divided into four groups, with each group undergoing one of the following treatments:

- 0 hours @ 200°C (as-received)
- 50 hours @ 200°C with ~800 Pa water vapor pressure
- 50 hours @ 300°C with ~800 Pa water vapor pressure
- 50 hours @ 400°C with ~800 Pa water vapor pressure

(Due to material limitations, the AC Sparkplug material was only subjected to the first two treatments.)

Table 1. EVALUATED MATERIALS

	Code	Manufacturer	Material	Process	Mole% Y <sub>2</sub> O <sub>3</sub>	Mean Grain Size (μm)
Japanese	KY	Kyocera	Z-201	Sintered	2.8	0.65
	TOSH	Toshiba	TASZIC	Sintered	2-3	0.49
	HIT	Hitachi	1985	Hot-Pressed (?)	2.0	0.37
	NGK	NGK Locke	Z-191	Sintered	3.0	0.18
	KS	Koransha	1986	Sintered	3.0	0.50
	KH	Koransha	1936	HIP'ed	3.0	0.41
Domestic	AC	AC Sparkplug	TZP-110	Sintered	2.6	0.80

Treatments were done in an autoclave apparatus consisting of a one liter pressure vessel, a furnace, temperature controller, timer, pressure gauge, and power supply. Bars were placed in the pressure vessel on a stainless steel wire mesh screen which minimized obstacles to heat



flow and maintained uniformity throughout the test. The pressure was kept constant by varying the amount of water placed in the vessel prior to the start of the test.

The bulk density was measured by the Archimedes technique. Room temperature strength was determined from a minimum of 10 bars for each material per condition (except where noted) using four-point bending, according to MIL-STD-1942 (MR), with inner and outer spans of 20 mm and 40 mm for the type "B" bars, and 10 mm and 20 mm for type "A", as well as a crosshead speed of 0.5 mm/min for both. The fracture surface of each bar was examined optically using a low magnification microscope in an attempt to determine the cause of failure. In several cases, a scanning electron microscope (SEM) was used to improve the chances of determining the flaw type.

The linear intercept method was used to determine the average grain size of the as-received material only. This method was applied to SEM micrographs of specimens which were thermally etched at 1450°C for 15 minutes. The presence and amount of zirconia phases in each material was determined using x-ray diffraction with  $\text{CuK}\alpha$  radiation over an angular range of  $25^\circ$  to  $40^\circ 2\theta$ . The surface-volume fraction of monoclinic and tetragonal-plus-cubic zirconia was calculated using the equations and constants in Reference 18, which take into account the difficulty in deconvoluting the tetragonal (101) and cubic (111) peaks.

## RESULTS

### Observations

After the three treatments, which included the water vapor, physical changes were quite obvious in four of the seven materials. All of the TOSH and all but two bars of the KY fractured into several pieces due to the 200°C treatment, as shown in Figures 1b and 2b. Subsequent treatments at 300°C and 400°C resulted in more severe degradation of all the bars of both of these TZPs (as shown in Figures 1c and 2c). Low power ( $\sim 25\times$ ) optical examination of the surfaces of the remaining pieces of each material showed extensive macrocracking, as shown in Figure 3. Micro- and macrocracking have been reported<sup>2,6,9,11,13</sup> to be common indicators of the occurrence of the  $t \rightarrow m$  transformation in this phenomenon.

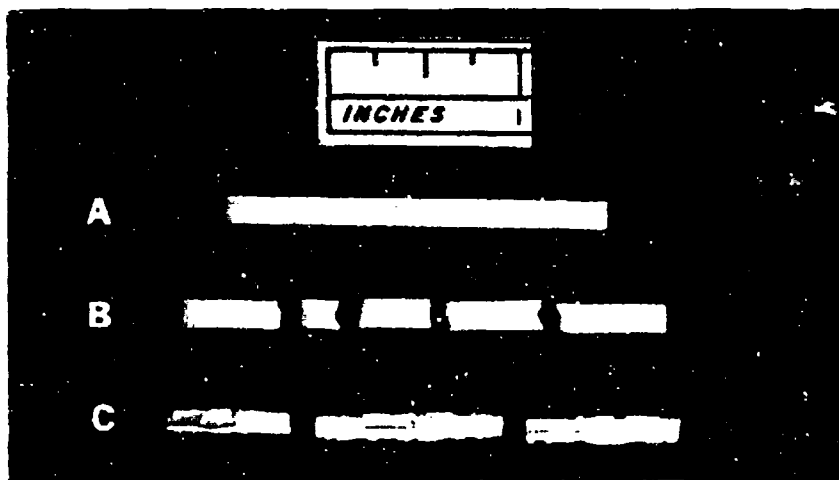


Figure 1. (a) TOSH in the as-received state, (b) after 50 hours at 200°C with  $\sim 800$  Pa water vapor pressure, and (c) after 50 hours at 300°C or 400°C with  $\sim 800$  Pa water vapor pressure.

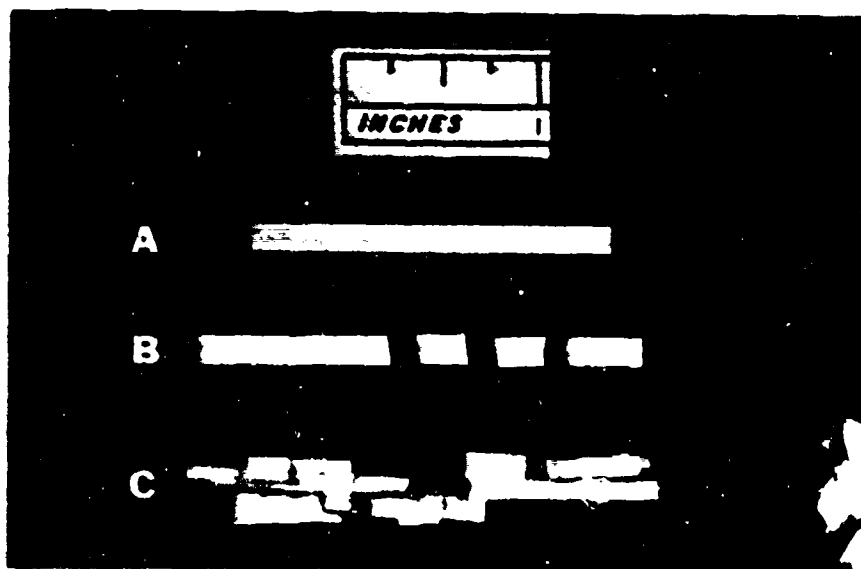


Figure 2. (a) KY in the as-received state, (b) after 50 hours at 200°C with ~800 Pa water vapor pressure, and (c) after 50 hours at 300°C or 400°C with ~800 Pa water vapor pressure.



Figure 3. Optical photograph of macrocracks on the surface of TOSH, AC, and KY TZPs.

One of the general observations of this degradation phenomenon is that the  $t \rightarrow m$  transformation and accompanying micro- and macrocracking initiate on the surface and proceed into the bulk. This implies that a larger volume of material will require longer exposure times before disintegration occurs than will the same material in a much smaller volume exposed to identical conditions. This is the case with the AC material. Both type "A" and "B" bars were exposed to 200°C with water vapor for 50 hours. The "A" bars completely disintegrated, as shown in Figure 4c, but the "B" bars, as shown in Figure 4a, remained intact.

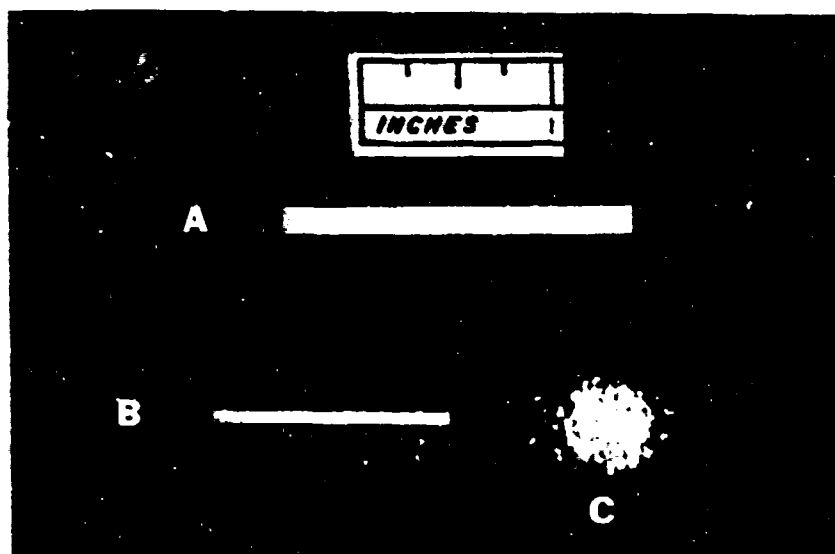


Figure 4. (a) AC type "B" bar in the as-received state and after 50 hours at 200°C with ~800 Pa water vapor pressure, (b) AC type "A" bar in the as-received state, and (c) after 50 hours at 200°C with ~800 Pa water vapor pressure.

The fourth material which underwent physical change was the HIT at 200°C with water vapor. As shown in Figure 5, entire sides of a bar spalled off in sheets of relatively uniform thickness. This spalling did not occur after the treatments at higher temperatures. The number of sides which spalled off varied with each bar. These sheets ranged in thickness from 250  $\mu\text{m}$  to 650  $\mu\text{m}$ . Schmauder and Schubert<sup>9</sup> and Yoshimura et al.<sup>13</sup> have seen large cracks running parallel to the surface of the specimen. It is believed that the crack separates the transformed material from the untransformed material. The former study measured the thickness of the degraded layer in a 2.0 mole% Y-TZP exposed for 1000 hours at 250°C in air at about 100  $\mu\text{m}$ . The difference in thickness and crack severity can be attributed to the presence of water vapor during the treatment used in the present study since it has been shown that the presence of water enhances the  $t \rightarrow m$  transformation.<sup>5,12-14,16</sup> The remaining Y-TZPs showed no signs of physical change.

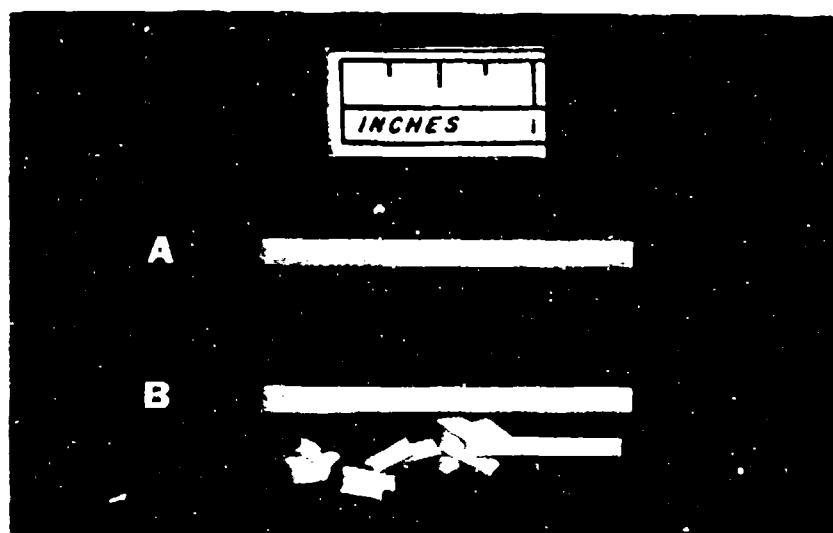


Figure 5. (a) HIT in the as-received state and after 50 hours at 300°C or 400°C with ~800 Pa water vapor pressure, and (b) after 50 hours at 200°C with ~800 Pa water vapor pressure.

## Density and Modulus of Elasticity

Figure 6 shows that the density of the TOSH, AC, and KY TZPs decreased between 2% and 6% due to the treatments. A fourth TZP, HIT, decreased -4% after the 200°C treatment, but only 1% and <1% after treatment at 300°C and 400°C, respectively. Watanabe et al.<sup>2</sup> have seen similar changes in density, however, this was seen in a 4 mole% yttria stabilized zirconia. The density of the remaining materials is unaffected by the treatments.

Corresponding to the density decrease is a proportional decrease in the modulus of elasticity (MOE) of the KY and HIT after the 200°C treatment, as shown in Figure 7. The MOE of all the TOSH and AC, and the KY after the 300°C and 400°C treatments could not be determined due to the severe degradation of the bars.

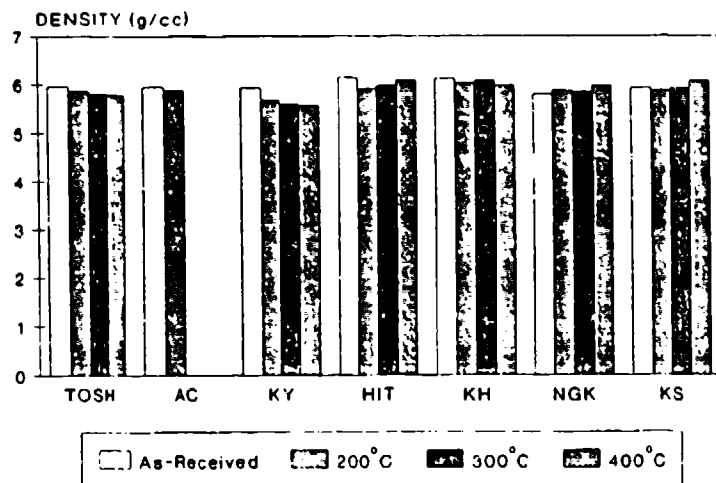


Figure 6. Change in density after 50 hours at temperature with ~800 Pa water vapor pressure.

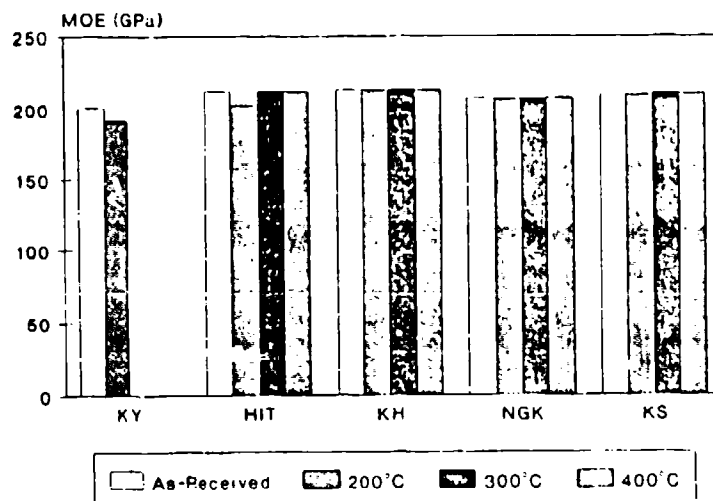


Figure 7. Change in modulus of elasticity after 50 hours at temperature with ~800 Pa water vapor pressure.

## Phase Stability

The change in surface m-ZrO<sub>2</sub> content with heat treatment temperature is shown in Figure 8. With the exception of NGK, all other TZPs showed tremendous increases in the surface m-ZrO<sub>2</sub> content. The TOSH, AC, and KY all had 200% increases in surface m-ZrO<sub>2</sub> after all heat treatment temperatures. HIT had an increase from 10% to almost 80% after the 200°C and 300°C treatment, but after the 400°C the m-ZrO<sub>2</sub> content increased to just over 20%. The KH also had a tremendous initial increase from ~15% to ~80% but the amount gradually decreased to 60% and 42% after the 300°C and 400°C treatments. The KS TZP showed a 500% to 700% increase after all treatments. Many investigators<sup>2-6,11-14,16,17</sup> have seen similar increases in the amount of monoclinic phase when zirconia stabilized with 2 to 4 mole% yttria is subjected to low temperature treatments (200°C to 400°C) in the presence of water.

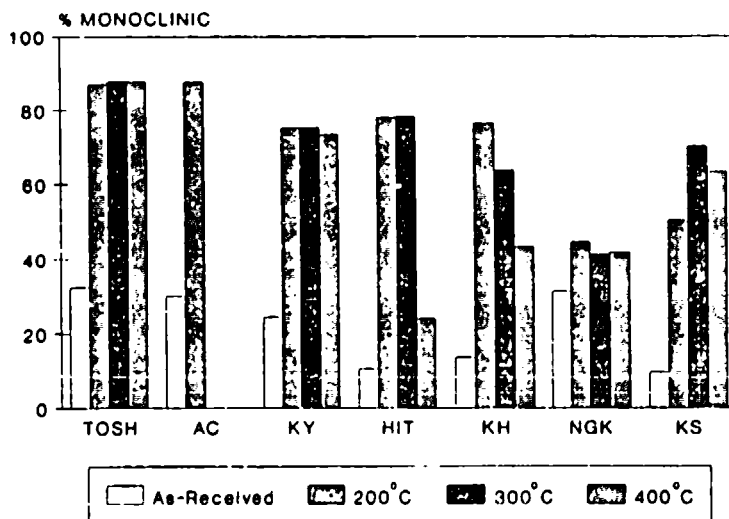


Figure 8. Change in surface monoclinic content after 50 hours at temperature with ~800 Pa water vapor pressure.

## Strength\*

Three of the five TZPs which could be used for flexure strength testing showed significant strength degradation as a result of the treatments, as shown in Figure 9. The strength of KY after the 200°C treatment was approximately 50% less than for the as-received condition. This amount of loss is similar to what has been seen for this material after exposure to 1000°C for 500 hours.<sup>19</sup>

The HIT and KH both showed a significant and in strength degradation in that the degradation was most severe during the 200°C treatment and gradually decreased as the treatment temperature increased. However, the degradation was more pronounced in HIT, as it lost 68%, 41%, and 16% of its room temperature strength with each increase in temperature, while the KH lost only 22%, 16%, and 8% for the same treatments, respectively.

\*Although size "B" bars of the AC material survived the 200°C treatment, a strength comparison could not be made since the as-received strength was determined with "A" size bars only. Nevertheless, the "B" bars were broken in four-point bending to determine the location of the failure origin and the depth of transformation.

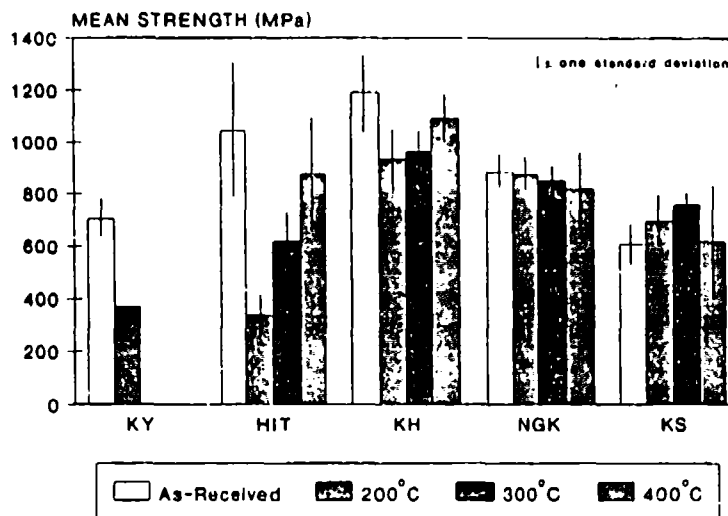


Figure 9. Change in room temperature strength after 50 hours at temperature with  $\sim 800$  Pa water vapor pressure.

The percentage of strength degradation reported here is in excellent agreement with that published by Watanabe et al.<sup>2</sup> and Sato et al.<sup>12,16</sup> for zirconias stabilized with 2 to 5 mole% yttria subjected to similar test conditions.

Although NGK and KS did show slight fluctuations in strength with the treatments, these changes were well within experimental error. This indicates the strength of both materials was essentially unaffected by the treatment.

### Fractography

Analysis of the fracture surface of the as-received bars of all the TZPs showed that the strength-limiting flaws tended to be porosity related; i.e., pore, porous region, etc., but other flaw types such as large grain(s), inclusions, and machining damage were also observed, as shown in Figure 10. Examination after treatments revealed that in several instances the flaw location, rather than the flaw type, changed.

In the AC material, as shown in Figure 11a, there is a surface layer which is believed to be m-ZrO<sub>2</sub> that has been transformed from the tetragonal phase due to the treatment. This m-ZrO<sub>2</sub> layer is approximately 100  $\mu$ m to 150  $\mu$ m deep around the entire bar. Other investigators<sup>4,6,12,13,16,17</sup> have also reported seeing these zones of transformation. The strength-limiting flaw is still porosity related, as shown in Figure 11b, but it is located in the t-ZrO<sub>2</sub> just inside the interface which separates the two phases. For the two KY bars which survived the 200°C treatment, the zone of transformation was  $\sim 300$   $\mu$ m deep and expanded to  $\sim 600$   $\mu$ m around the chamfers of the bar, as shown in Figure 12. Macrocracking can be seen to penetrate  $\sim 50$   $\mu$ m into the m-ZrO<sub>2</sub> zone. The microcracking in conjunction with the large m-ZrO<sub>2</sub> transformation zone is the probable cause of failure since there is no evidence of a single-strength limiting flaw on the fracture surface.

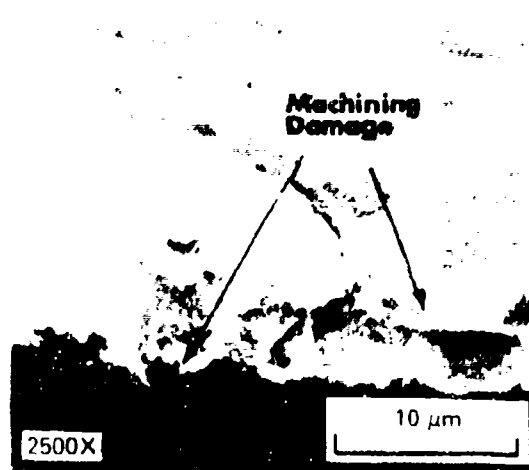
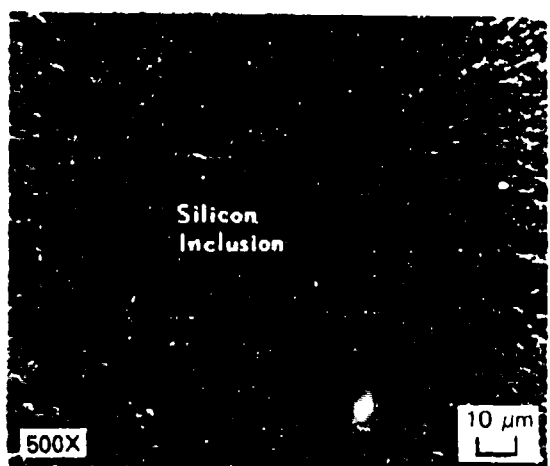
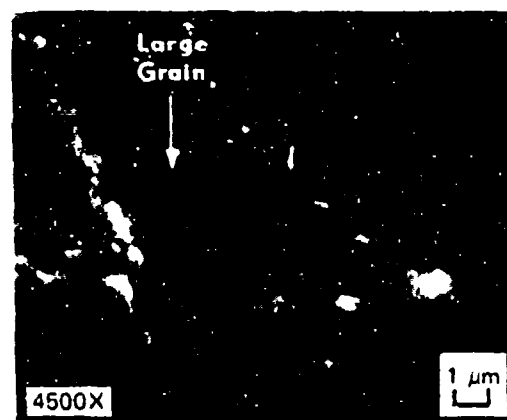
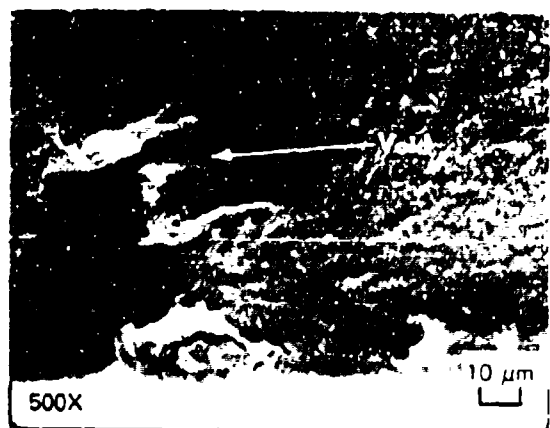
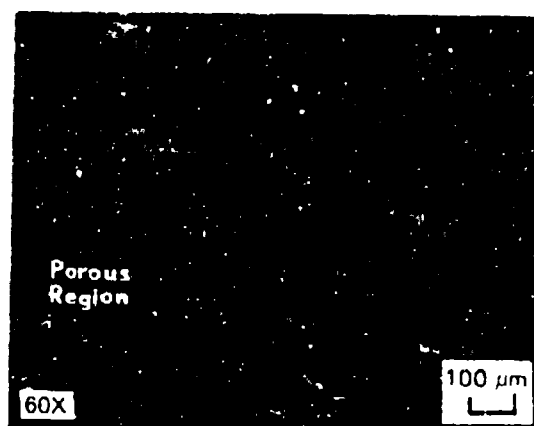
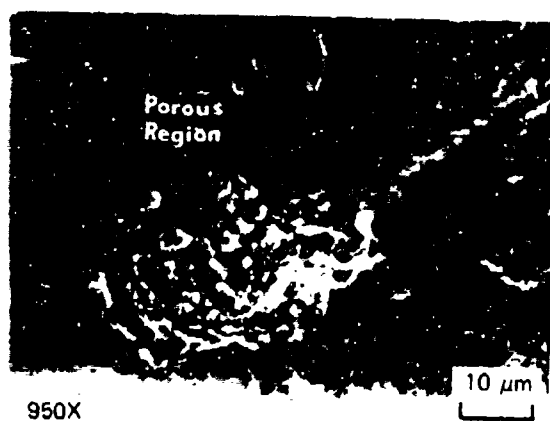


Figure 10. Strength limiting flaws in the as-received TZP materials.

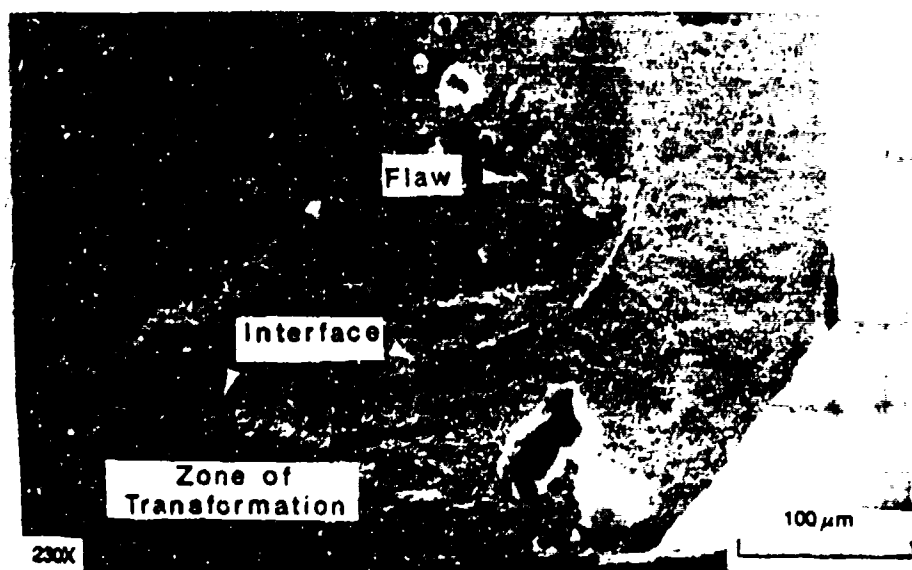


Figure 11a. SEM photo of the fracture surface of an AC bar after 50 hours at 200°C with ~800 Pa water vapor pressure showing the depth of the transformation zone and the flaw location.



Figure 11b. SEM photo of the strength limiting flaw, porous region, in the same AC bar.

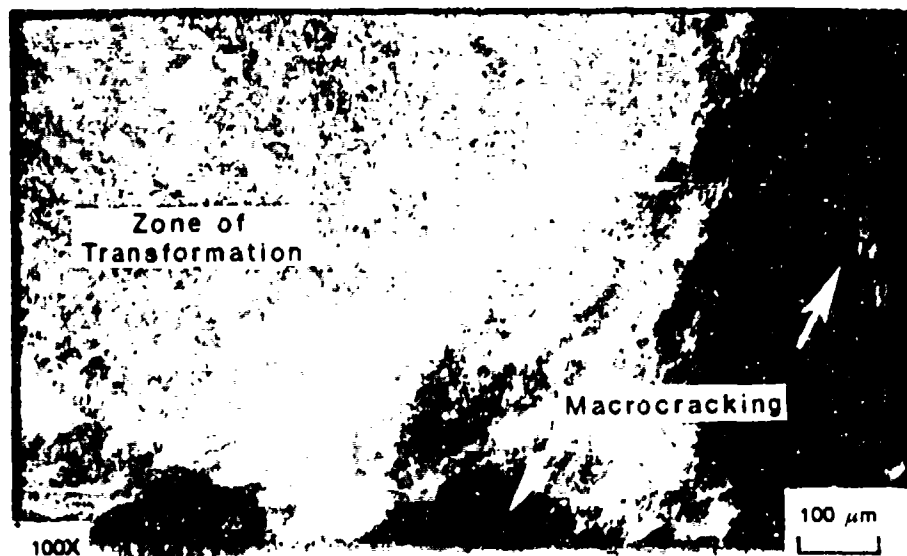


Figure 12. SEM photo of the fracture surface of a KY bar after 50 hours at 200°C with ~800 Pa water vapor pressure showing the depth of the transformation zone and the associated macrocracking.



Fractography of the HIT after the 200°C treatment was difficult because it was very easy to remove sheets of material which had not spalled off during the treatment. Figure 13a is a low-power (~20X) photo of the fracture surface of a bar which shows three sides remaining intact. The interface between the untransformed t-ZrO<sub>2</sub> and the m-ZrO<sub>2</sub> surface layer is easily seen, but the strength-limiting flaw is not obvious. Figure 13b shows a pore located at the junction of the t-ZrO<sub>2</sub> material and two sections of the m-ZrO<sub>2</sub> layer; one of which is no longer attached to the bar. Cracks can be seen running parallel to the interface and the higher magnification photo, Figure 13c, shows the buildup of layers within the m-ZrO<sub>2</sub> zone and extensive microcracking. Examination after the subsequent treatments showed a large reduction in the size of the transformation zone to ~50 μm after the 300°C treatment, and to 15 μm to 20 μm after the 400°C treatment, as shown in Figures 13d and 13e. There is microcracking associated with the m-ZrO<sub>2</sub> zone in Figure 13d, but none is evident in Figure 13e.

The KH material shows a very small transformation zone of 10 μm to 20 μm after the 200°C and 300°C treatments, but no discernible zone after the 400°C treatment, as shown in Figures 14a through 14c. There is evidence of microcracking in Figure 14a. Examination of the fracture surfaces after the 400°C treatment indicates that the inherent flaws now dominate, as is the case in Figure 14c in which Al inclusions are the strength-limiting flaw for this bar.

Fractography of the NGK and KS TZPs showed no signs of a zone of transformation after any of the treatments. Furthermore, the same flaw types limit the as-received strength and the post heat treatment strengths.

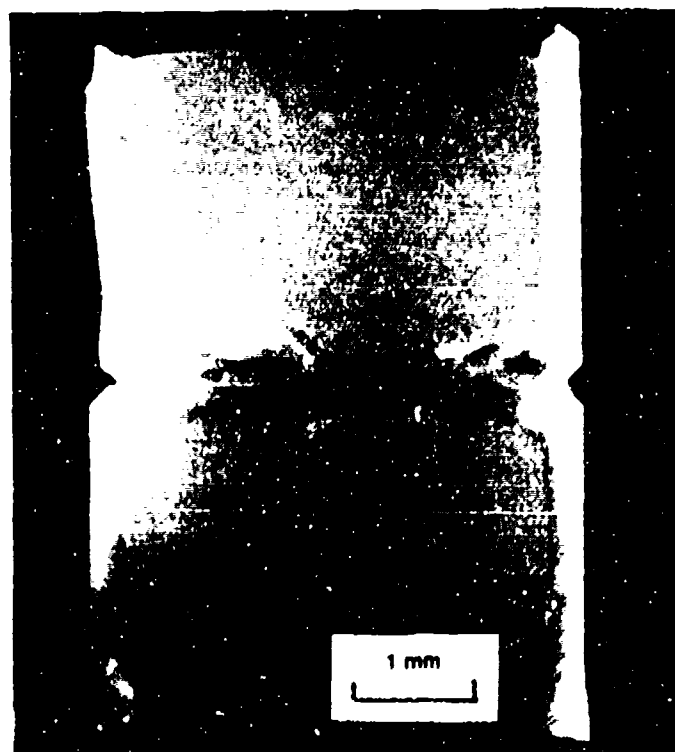


Figure 13a. Optical photograph of a HIT bar after 50 hours at 200°C with ~800 Pa water vapor pressure.

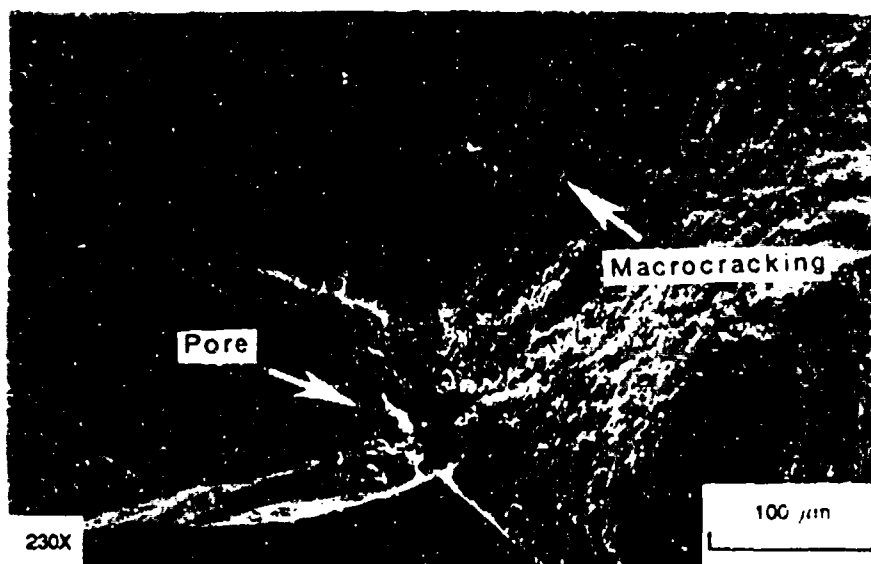


Figure 13b. SEM photo of a HfT bar after 50 hours at 200°C with ~800 Pa water vapor pressure showing the flaw location and the interface between the transformation zone and the untransformed zirconia.

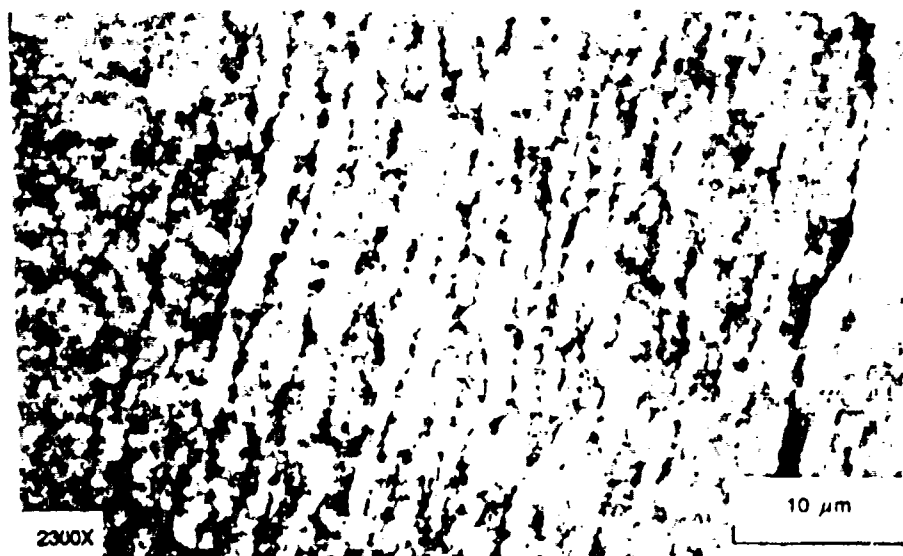


Figure 13c. SEM photo of the layered structure of the transformation zone.

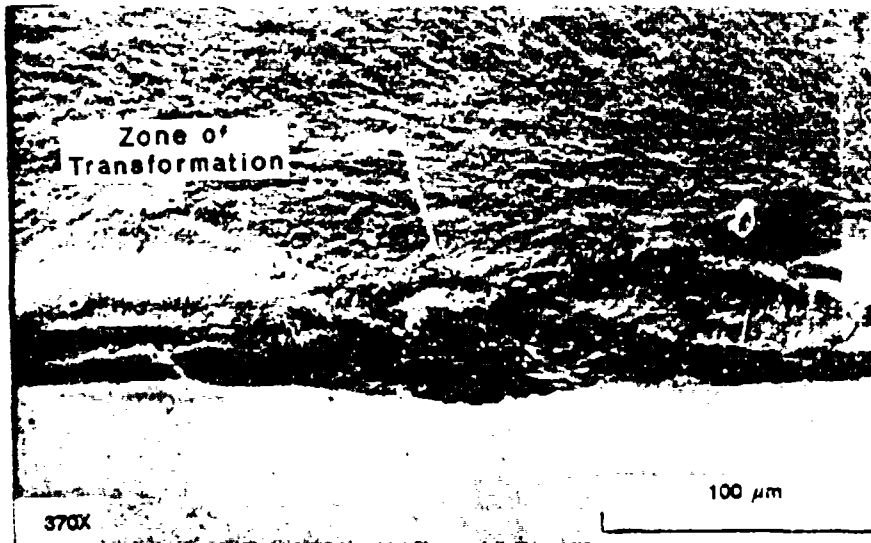


Figure 13d. SEM photo of the fracture surface of HIT after 50 hours at 300°C with ~800 Pa water vapor pressure.

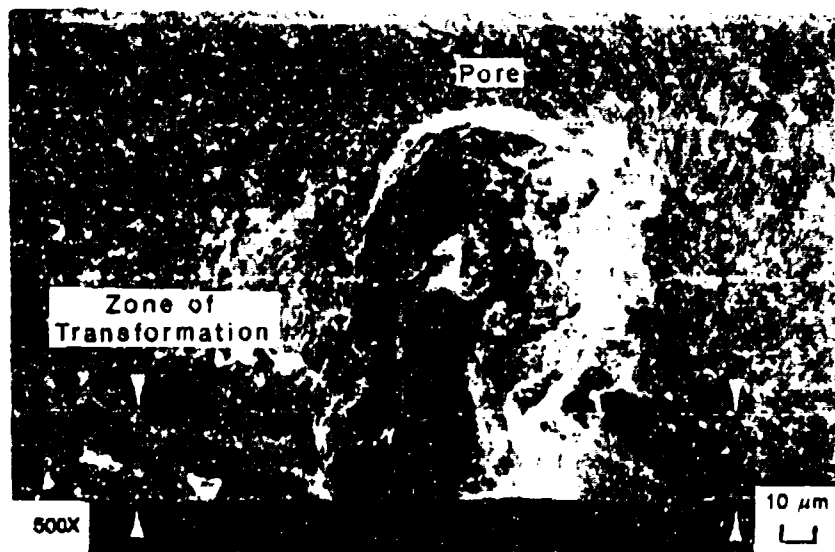


Figure 13e. SEM photo of the fracture surface of HIT after 50 hours at 400°C with ~800 Pa water vapor pressure.

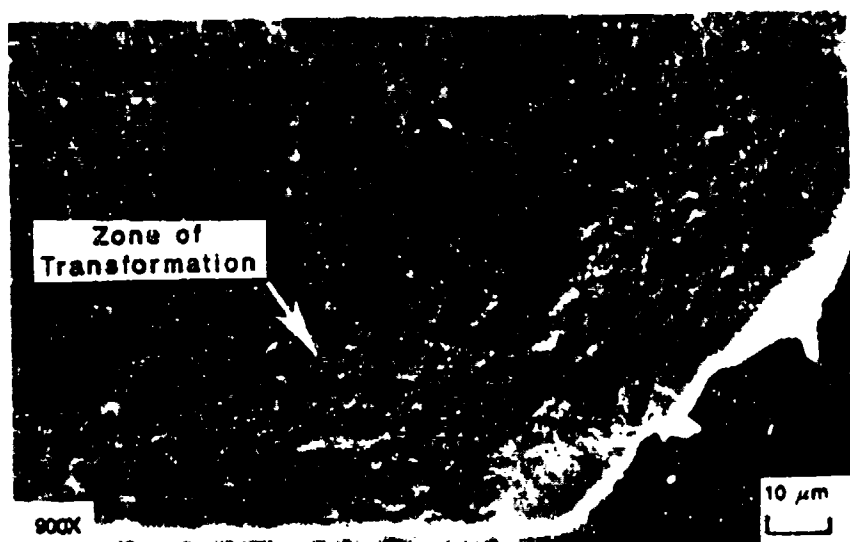


Figure 14a. SEM photo of the fracture surface of KH after 50 hours at 200°C with ~800 Pa water vapor pressure.

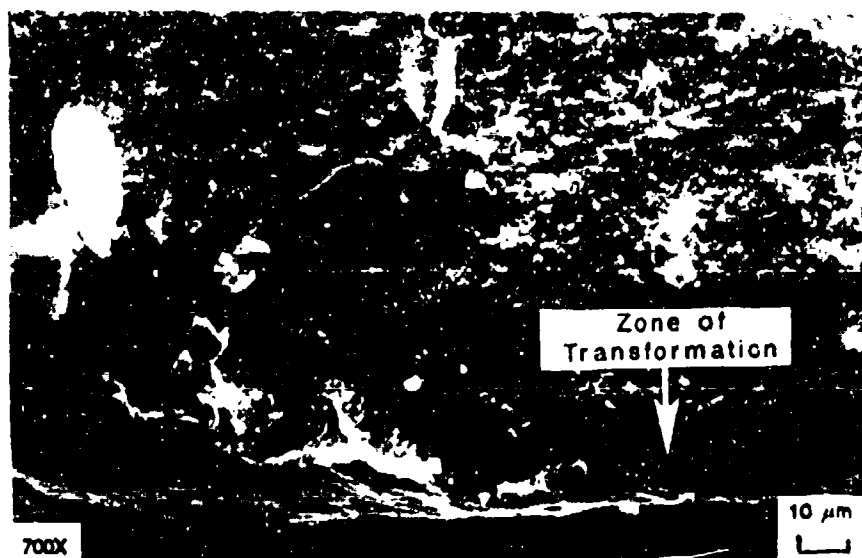


Figure 14b. SEM photo of fracture surface of KH after 50 hours at 300°C with ~800 Pa water vapor pressure.



Figure 14c. SEM photo of the fracture surface of KH after 50 hours at 400°C with ~800 Pa water vapor pressure.

## DISCUSSION

From the results reported here, and those previously referenced, the surface layers seen during fractographic analysis are clearly m-ZrO<sub>2</sub>, which has transformed from t-ZrO<sub>2</sub> as a result of the heat treatments. The ability of the seven TZP materials to maintain their properties is directly related to the depth of the transformation zone and the presence of micro- and macrocracking. TZPs with large zones of transformation (>600  $\mu$ m) and extensive cracking showed large property losses, and, in most instances, catastrophic degradation of the material; i.e., TOSH, KY, and the type "A" bars of AC.

The HIT and KH TZPs also had significant property losses after these treatments, but the most severe losses came only after the 200°C treatment. In the case of HIT, where thermal spalling occurred due to the 200°C treatment, it is possible that this would have occurred at the other treatment temperatures if the exposure time and/or water pressure was increased, but further testing would be required to confirm this.

Other TZPs; i.e., NGK and KS, that had very little, if any, zone of transformation were able to retain their properties after exposure even though the m-ZrO<sub>2</sub> content increased. This indicates that though these materials are not totally resistant to the t  $\rightarrow$  m transformation, they can tolerate slight crystallographic changes on the surface without an adverse effect on their properties. This may not hold true if the material is subjected to a longer or more severe heat treatment.

The performance of these TZPs after exposure to low temperatures in the presence of water can be summarized by examining the grain size and yttria content of each. Several references<sup>2,4,6-9,11,16</sup> have shown that an increase in the yttria content, a decrease in the grain size, or a combination of both will result in a TZP with superior resistance to low temperature degradation.

Lange reported that the retention of the tetragonal phase during processing is dependent on a combination of grain size and yttria content.<sup>20</sup> By plotting the grain size versus mole% yttria, Lange was able to define a critical grain size ( $D_c$ ) necessary for the retention of >90% of the tetragonal phase for a given yttria content during normal processing. The general trend being that as the amount of yttria additive decreased, so must  $D_c$ . Above  $D_c$  for a given yttria content, the retention of the tetragonal phase becomes increasingly more difficult. The same relationship also seems to apply to the low temperature degradation phenomenon.<sup>2,6,11</sup> Figure 15 summarizes the data from these investigations.

By adding the data from the seven TZPs examined in this study to Figure 15, it is clear that the KH, KS, and NGK TZPs should be the most resistant to low temperature degradation. This is, in fact, the case. All three TZPs are in excellent agreement with the findings of References 2, 6, and 11 for TZPs containing 3.0 mole% yttria. Since the NGK is significantly below the curves from these references, as shown in Figure 15, it is expected that the material would still be very resistant to low temperature degradation even if the severity of the treatment conditions were increased. To increase the resistance to low temperature degradation of the remaining four TZPs, changes must be made in the processing of each material. The TOSH, AC, and KY could be improved by adjusting the sintering schedule to reduce the possibility of grain growth, or by using a hot-pressing or hot-isostatic-pressing technique instead of sintering. Minor increases in the yttria content, to almost 3.0 mole%, may also correct this problem. Since the HIT is produced by hot pressing, and it already has a very fine

grain size, it isn't likely that changes in the processing technique would be sufficient to reduce the effects of low temperature degradation; however, an increase in the yttria content would appear to be more beneficial.

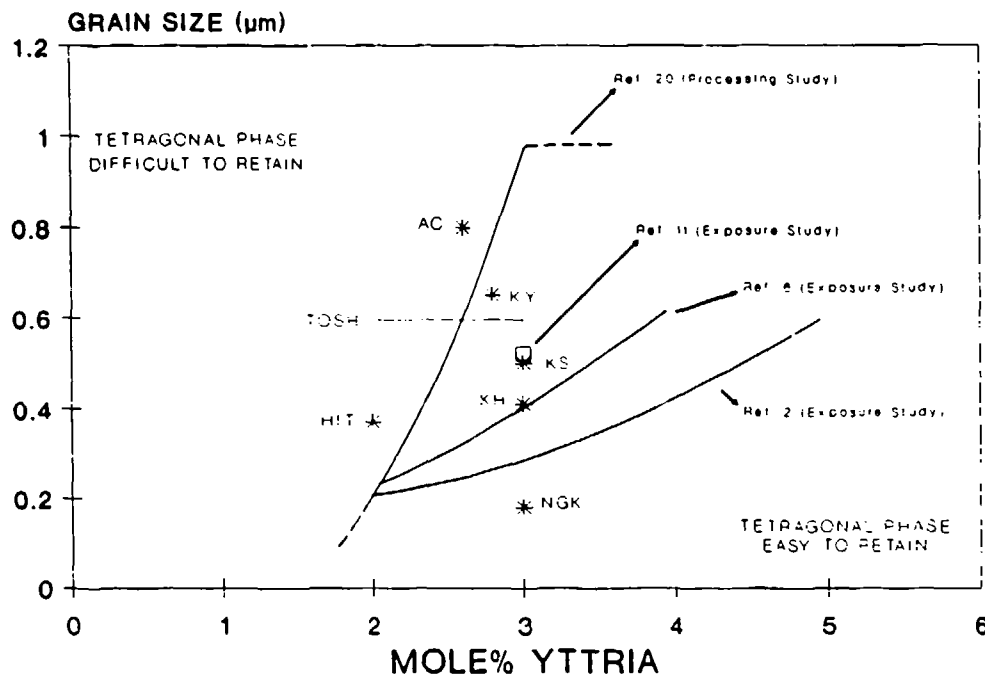


Figure 15. Grain size versus mole% yttria for the seven TZPs examined in this study and the data from References 2, 6, 11, and 20. The curves approximate the boundary between the ability to retain the tetragonal phase. Data from the present study is shown as points for comparison.

The present work and the studies referenced within show that a fine-grained microstructure is important if a Y-TZP material is to resist low temperature degradation between 200°C and 400°C. However, previous work by the author<sup>19</sup> on these same Y-TZPs showed that the fine-grained Y-TZPs have lower fracture toughness values than their coarse-grained counterparts. (The exception to this is NGK which has a relatively high toughness due to microstructural tailoring.) As a result, this trade-off between resistance to low temperature degradation and fracture toughness must be considered when examining a Y-TZP for structural applications.

### CONCLUSIONS

The first five general observations outlined in the Introduction were observed. The final one was not observed since none of the TZPs were exposed to low temperatures in the absence of water or water vapor.

One of the seven TZPs (NGK) examined was unaffected by the low temperature treatments in the presence of water vapor.

Two of the remaining TZPs (KS and KH) were affected by the treatments, the m-ZrO<sub>2</sub> content increased and the properties decreased slightly, but the extent of degradation was minimal.

A fourth TZP (HIT) had significant degradation after the 200°C treatment due to spalling of the sides of the bar. This effect was not present when the treatment temperature increased.

The final three TZPs (TOSH, AC, and KY) experienced catastrophic degradation after all treatments due to the spontaneous tetragonal-to-monoclinic transformation.

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# APPENDIX. ZIRCONIA DATA AFTER LOW TEMPERATURE EXPOSURE

		Material	KY	AC	TOSH	HIT	KS	KH	NGK
Property		Units							
Density:	Company Listing	g/cc	5.9	NDA	6.05	6.08	6.05	NDA	5.91
	As-Received		5.93	5.95	5.96	6.15	5.93	6.13	5.80
	50 Hrs @ 200°C w/H <sub>2</sub> O		5.68	5.87	5.86	5.92	5.87	6.03	5.88
	50 Hrs @ 300°C w/H <sub>2</sub> O		5.59	*****	5.81	5.98	5.92	6.08	5.84
	50 Hrs @ 400°C w/H <sub>2</sub> O		5.56	*****	5.78	6.09	6.07	5.97	5.97
Sonic MOE:	Company Listing	GPa	206	NDA	180	209	NDA	NDA	205
	As-Received		201	204	200	213	210	214	208
	50 Hrs @ 200°C w/H <sub>2</sub> O		192	201	-----	203	210	214	207
	50 Hrs @ 300°C w/H <sub>2</sub> O		-----	*****	-----	213	211	214	207
	50 Hrs @ 400°C w/H <sub>2</sub> O		-----	*****	-----	212	211	214	208
MOR (4-Point): (Mean)	Company Listing	MPa	980a	NDA	900a	1000	1100	NDA	1020a
	As-Received		704	722	587	1045	608	1192	884
	50 Hrs @ 200°C w/H <sub>2</sub> O		369	-----	-----	337	696	935	876
	50 Hrs @ 300°C w/H <sub>2</sub> O		-----	*****	-----	618	759	963	851
	50 Hrs @ 400°C w/H <sub>2</sub> O		-----	*****	-----	876	619	1094	817
Standard Deviation:	Company Listing	MPa	NDA	NDA	NDA	NDA	NDA	NDA	NDA
	As-Received		75	70	96	265	75	140	65
	50 Hrs @ 200°C w/H <sub>2</sub> O		3	19	-----	77	94	115	60
	50 Hrs @ 300°C w/H <sub>2</sub> O		-----	*****	-----	112	45	80	58
	50 Hrs @ 400°C w/H <sub>2</sub> O		-----	*****	-----	211	215	86	139
Grain Size: (Mean)	Company Listing	μm	NDA	NDA	0.5	0.3	NDA	NDA	NDA
	As-Received		0.65	0.80	0.49	0.37	0.50	0.41	0.18

KY - Kyocera Z-201  
 AC - AC Sparkplug TZP-110  
 TOSH - Toshiba "TASZIC"  
 HIT - Hitachi  
 KS - Koransha "sintered"  
 KH - Koransha "HIP'ed"  
 NGK - NGK-Locke Z-191

NDA - no data available  
 a - believed to be 3-point bend results  
 \*\*\*\*\* - not used in this test



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